

## CLAIMS

## {Claim(s)}

[Claim 1] The manufacturing method of the chlorination alkyl to which use the alkyl dimethylamine of the carbon numbers 6-20 of an alkyl group for as a reaction accelerator, and introduce a gas hydrochloric acid in the system of reaction, and it is made to react in case alcohol and a hydrochloric acid are made to react and chlorination alkyl is manufactured.

[Claim 2] The manufacturing method according to claim 1 which removes a reaction accelerator by the slice.

[Claim 3] The manufacturing method according to claim 1 or 2 it is twice [0.005-0.8 mol] whose addition of a reaction accelerator of this to alcohol.

## DETAILED DESCRIPTION

## {Detailed Description of the Invention}

## {0001}

[Industrial Application] This invention relates to the approach of being high yield and manufacturing the chlorination alkyl of a high grade economically.

## {0002}

[Description of the Prior Art] As a manufacturing method of chlorination alkyl, alcohol and hydrogen chloride gas are continuously introduced into a reaction vessel, and the method of using amines, such as a pyridine and tributylamine, for the approach of using a zinc chloride for the approach of making the chlorination alkyl to generate, a by-product, water, and superfluous hydrogen chloride gas distilling continuously, and JP,5-57973,B, as a catalyst, and JP,48-26685,B, as a catalyst is indicated by JP,6-1729,A. Although characterized by for these approaches supplying continuously the alcohol and hydrogen chloride gas which are reaction mixture, and separating a product continuously, it is not suitable for manufacture of the chlorination alkyl which has alkyl chain length's long high-boiling point, and many amounts of reaction by-products are also accepted.

[0003] Moreover, since productivity is sacrificed for this approach and it uses distillation generation for separation of a product, its load to a manufacturing facility is also large [in order to make hydrogen chloride capacity to be used into the minimum, the approach of using a lot of amines or its hydrochlorides is indicated by JP,49-34646,B, but]. Although the approach using the reaction accelerator which has a cyclohexane ring is indicated by JP,56-53289,B and JP,61-28649,B and the high selection-reaction is realized, since these approaches use a lot of reaction accelerators or use the hydrochloric-acid water solution, they have the trouble that productivity is inferior.

[0004] Furthermore, although the approach to which alcohol and a hydrochloric acid are made to react under pressurization is indicated by JP,58-144328,A, since it is expected that the facility which a pressurization reaction takes turns into a heavy facility, there is a problem economically.

[0005] The technical problem of this invention solves the above troubles, and it is in offering the approach of being high yield and manufacturing the chlorination alkyl of a high grade economically.

## {0006}

[Means for Solving the Problem] In case this invention makes alcohol and a hydrochloric acid react and manufactures chlorination alkyl, it is the manufacturing method of the chlorination alkyl to which use the alkyl dimethylamine of the carbon numbers 6-20 of an alkyl group for as a reaction

accelerator, and introduce a gas hydrochloric acid in the system of reaction, and it is made to react.

[0007]

[Embodiment of the Invention] As raw material alcohol used for this invention, the straight chain or branched chain alcohol of carbon numbers 6-24 especially the saturation of 8-24, or partial saturation is desirable.

[0008] From a viewpoint that the alkyl dimethylamine used as a reaction accelerator by this invention obtains the chlorination alkyl of a high grade, and raises productivity, the carbon numbers of an alkyl group are 6-20, and 8-18 are desirable. The addition of this reaction accelerator has desirable 0.005-0.5-mol twice to alcohol, and its 0.01-0.3-mol twice are still more desirable.

[0008] What distilled for the reaction of this invention, gas hydrochloric acid, i.e., hydrochloric-acid water solution, and was made into the gas is introduced. As for a superfluous hydrochloric acid, it is desirable to install a hydrochloric-acid absorption tower after a reaction vessel, and to collect as a hydrochloric-acid water solution.

[0010] The reaction temperature of this invention has desirable 120-180 degrees C, and its 130-160 degrees C are still more desirable. As for reaction pressure, it is desirable to carry out by ordinary pressure.

[0011] By putting after reaction termination, carrying out a slice, or putting after adding water and carrying out a slice, the reaction accelerators of this invention can be collected as an alkyl dimethylamine hydrochloride water solution, and the next reaction can be supplied with them as it is. Moreover, the water at this time is removed in a system-of-reaction temperature up at the outside of a system.

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[Example] The temperature up was carried out to 130 degrees C, teaching and stirring the dodecyl alcohol 1000g and 0.2 mol twice (alcohol for a raw material) of dimethyl octyl amines in the glass reaction container which attached example 1 fractionating tower. Then, the gas hydrochloric acid was introduced in the system of reaction at the twice (alcohol for a raw material) as many 0.4-mol/h rate as this. The gas chromatography analyzed the reduction rate of alcohol and the stop reaction was ended for installation of hydrochloric acid gas in the place where the amount of unreacted alcohol became 0.1%. The time amount to reaction termination is shown in Table 1 after hydrochloric-acid-gas installation initiation.

[0013] Moreover, the various reaction accelerators shown in Table 1 instead of a dimethyl octyl amine were made to react to \*\*\*\*\* and this appearance which are shown in Table 1 as a comparison. The result was also shown in Table 1.

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[0015] The result of Table 1 shows advancing a reaction very efficiently, even when a dimethyl octyl amine is little.

Another example of a reaction accelerator is a dimethyl octyl amine as 0.1 mol.

twice (alcohol for a raw material) -- it used and the same reaction as an example 1 was performed except making reaction temperature into 140 degrees C. A slice and rinsing removed the reaction accelerator after reaction termination, and chlorination dodecyl was obtained. Moreover, as a comparison, the zinc oxide (1-% of the weight pair raw material alcohol) was used instead of the dimethyl octyl amine, it reacted similarly, the zinc oxide was separated by distillation after that, and chlorination dodecyl was obtained. These results are shown in Table 2.

[0017]

[Table 2]

原料	反応時間 (時間)	変化率 <sup>ア</sup> (%)	副生物 (%)	未反応 率 <sup>ア</sup> (%)
本発明例 Y-288127-23	0.019	10	22.1	<0.1
	0.02	7	22.0	<0.1
	0.18	4	22.0	<0.1

[0018]\* : from the result of the table 2, such as the 2nd class chlorination alkyl, even if a by-product does not use the large distillation process of a load in facility, it can do separation easily, and it understands that product purity is also very high for a dimethyl octyl amine.

[0019] The same reaction as an example 1 was performed using the raw material alcohol and the reaction accelerator which are shown in example 3 table 3 except making reaction temperature into 165 degrees C, and making a reaction accelerator addition into 0.05-mol twice. A slice and rinsing removed the reaction accelerator after reaction termination, and chlorination alkyl was obtained. These results are shown in Table 3.

[0020]

[Table 3]

原料	反応時間 (時間)	変化率 <sup>ア</sup> (%)	副生物 (%)	未反応 率 <sup>ア</sup> (%)
本発明例 Y-288127-23	0.019	10	22.1	<0.1
	0.02	7	22.0	<0.1
	0.18	4	22.0	<0.1

[0021] The result of Table 3 shows that the reaction accelerator of this invention promotes a reaction efficiently.

[0022] The same reaction as an example 1 was performed except example 4 reaction temperature changing the addition of 180 degrees C and a reaction accelerator, as shown in Table 4. A slice and rinsing removed the reaction accelerator after reaction termination, and chlorination dodecyl was obtained. These results are shown in Table 4.

[0023]

[Table 4]

	反応促進剤	添加量 (GK倍率780-8)	反応時間 (時間)	変化率 <sup>ア</sup> (%)	副生物 (%)	未反応 率 <sup>ア</sup> (%)
本発明例 Y-288127-23	0.019	10		22.1	<0.1	<0.1
	0.02	7		22.0	<0.1	<0.1
	0.18	4		22.0	<0.1	<0.1

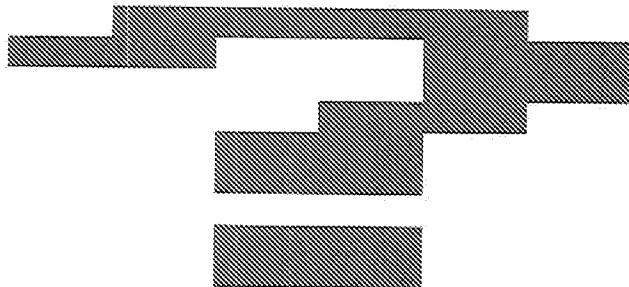
[0024] The result of Table 4 shows that the reaction accelerator of this invention promotes a reaction efficiently also with a small addition.

[0025] the alkyl dimethylamine or the comparison article of this invention which

shows to hexadecyl alcohol as example 5 raw-material alcohol, and is shown in Table 5 as a reaction accelerator -- 0.1-mol twice (alcohol for a raw material) -- it used and the same reaction as an example 1 was performed except making reaction temperature into 145 degrees C. These results are shown in Table 5.

[0026]

[Table 5]



[0027] The result of Table 5 shows that the reaction accelerator of this invention shows high performance compared with other amine compounds.